

# Optimizing Heat Treatment Parameters for 3rd Generation AHSS Using an Integrated Experimental-Computational Framework

**XIAOHUA HU**

X SUN<sup>1</sup>, KS CHOI<sup>1</sup>, G CHENG<sup>1</sup>

J MUELLER<sup>2</sup>, JG SPEAR<sup>2</sup>, DK MATLOCK<sup>2</sup>, E DE MOOR<sup>2</sup>

<sup>1</sup>PACIFIC NORTHWEST NATIONAL LABORATORY  
RICHLAND, WA, USA

<sup>2</sup>ADVANCED STEEL PROCESSING AND PRODUCTS RESEARCH CENTER (ASPPRC),  
COLORADO SCHOOL OF MINES, GOLDEN, CO, USA

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## Timeline

- Project start date: FY16
- Project end date: FY19
- Percent complete: 25%

## Budget

- Total project funding
  - DOE share: \$1,600k
  - ASPPRC in-kind: \$400k
- Funding for FY 2017:
  - DOE: \$369k
  - In-kind: \$100k

## Barriers

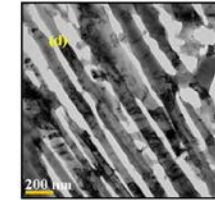
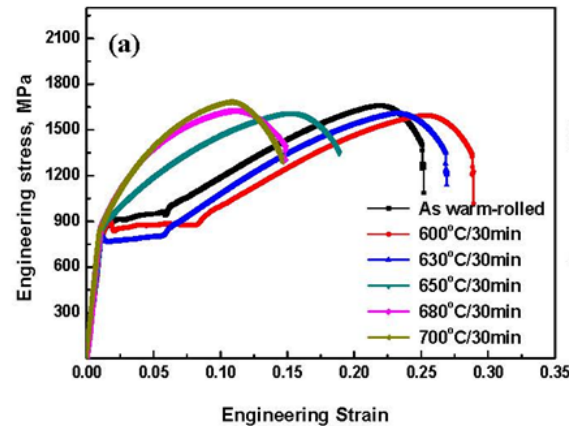
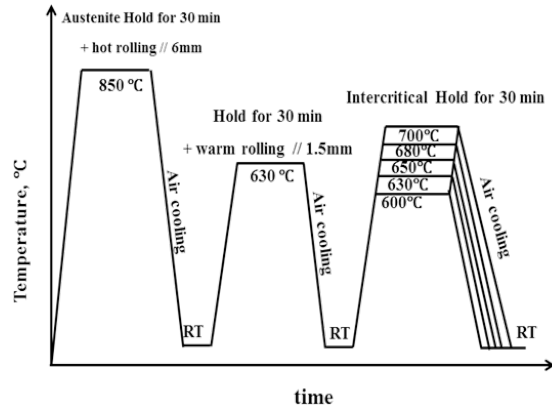
- Typical development to deployment cycle of 3<sup>rd</sup> GEN AHSS is very long
- Traditional experimental heat treatment and characterization techniques take too long for the development of medium Mn steels
- Lack of fundamental and quantitative understanding between alloying content, annealing parameters, austenite volume fraction and associated mechanical properties

## Partners

- Advanced Steel Processing and Products Research Center (ASPPRC)
- Colorado School of Mines (CSM)
- Advanced Photon Source (APS), Argonne National Lab (ANL)

# Traditional Experimental-based Med-Mn Steel Development

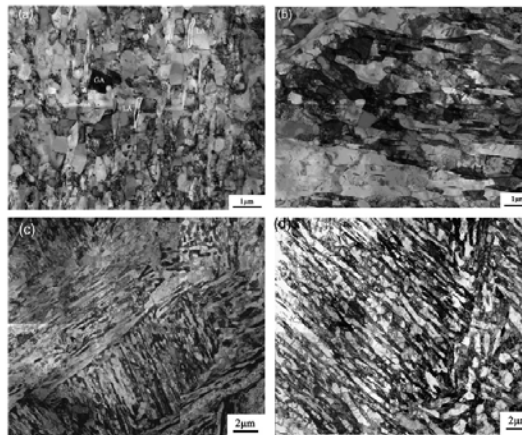
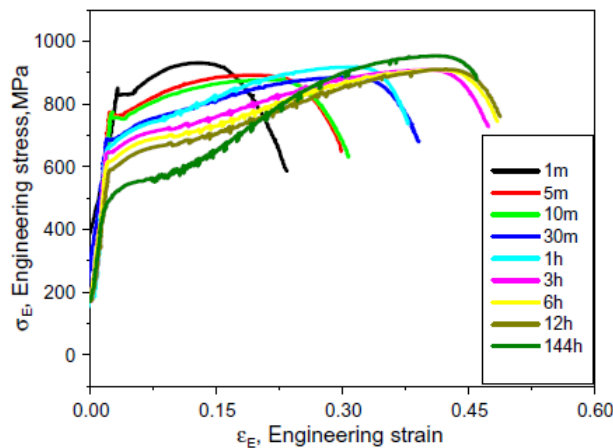
## ► Fe-7.9Mn-0.14Si-0.05Al-0.07C Steel



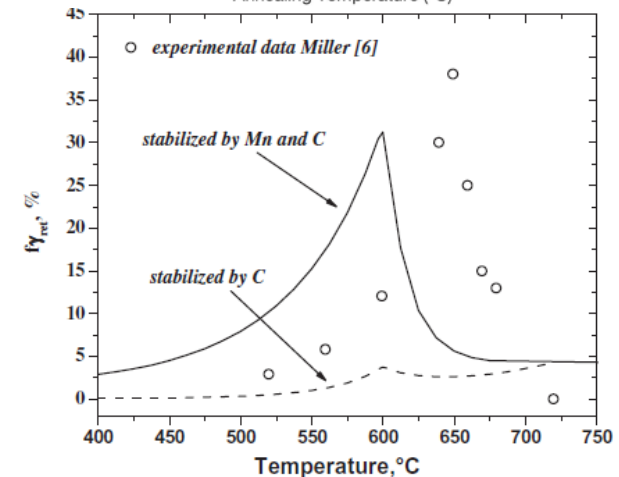
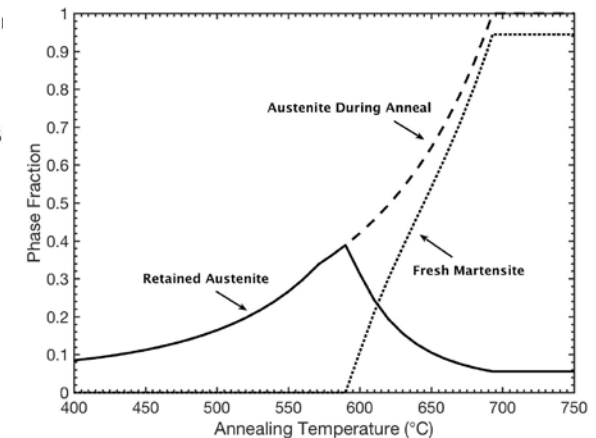
Zhao, et al., *Materials* 2014, 7

## ► Fe-5Mn-0.2C Steel

■ Austenization (AUS) + Intercritical annealing (IA) at 650 °C for up to 144h

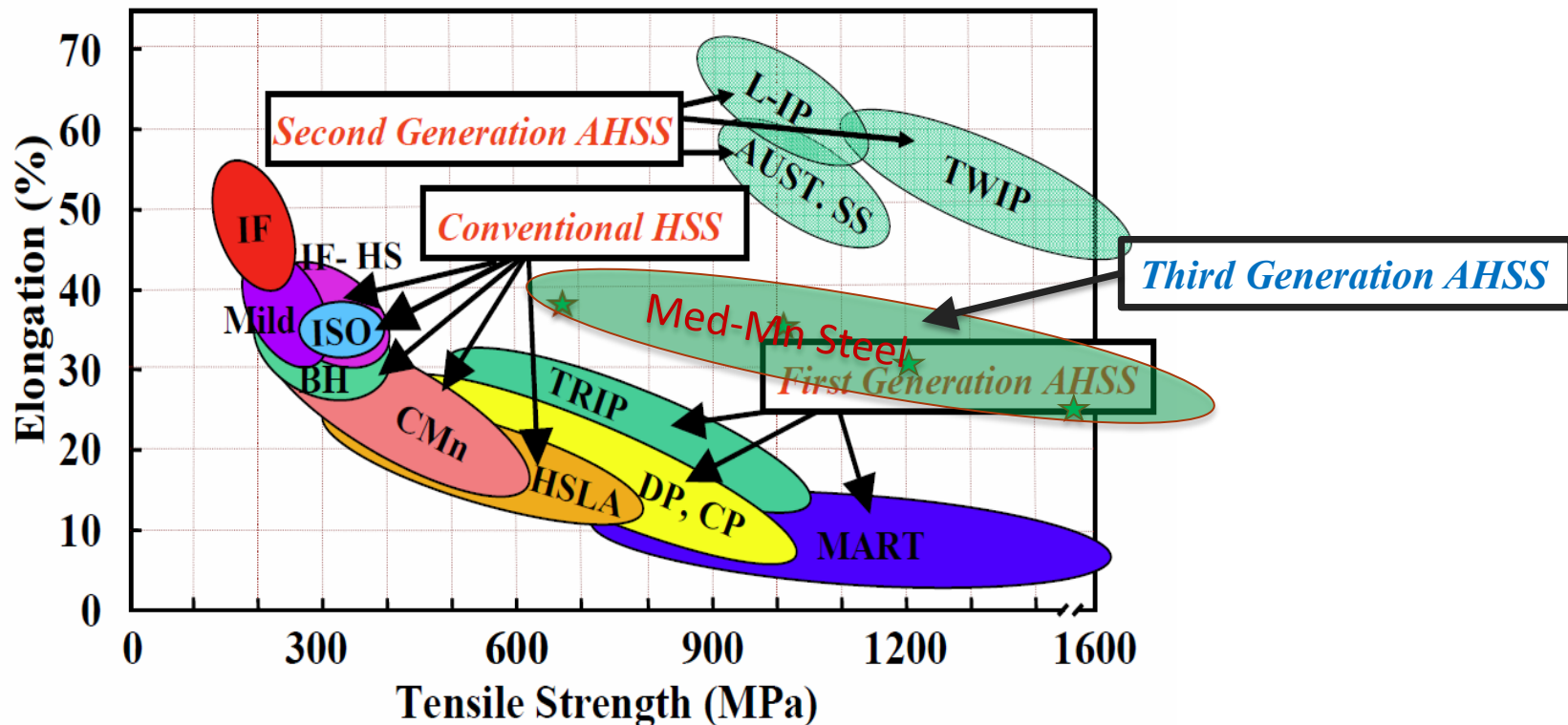


Luo, et al., *Acta Mat*, 2011.



# Relevance and Project Objectives

- ▶ Help steel producers & users to accelerate the development to deployment cycle of cost-effective 3<sup>rd</sup> generation advanced high strength steels (3G AHSS) with an integrated experimental and computational framework to meet DOE VTO MYPP targets and goals.



## ► Task 1 Conventional Experimental Characterization

- Microscopic characterization of retained austenite (RA) volume fraction, C and Mn content in different phases.
- Indentation test for phase property characterization.
- Macroscopic tests to determine tensile properties.

## ► Task 2 Advanced HEXRD in-situ characterization

- In situ HEXRD measurement of austenite formation kinetics at different annealing temperature.
- Estimate C, Mn content of different phases with changes of lattice parameters during in-situ high temperature HEXRD IA test.
- Perform in-situ HEXRD tensile test to determine austenite stability and individual phase properties under different IA conditions.

## ► Task 3 New models and integrated modeling framework development

- Develop phase-field based model with the capability to model austenite phase nucleation, transformation and C, Mn diffusion.
- Phase field predictions of austenite volume fraction, C and Mn distribution in the evolved microstructure under different annealing temperature and soaking time.
- Predict austenite stability with free energy calculation with various C and Mn content.
- Predict macroscopic tensile properties with the microstructure-based model and measured phase properties.

# Milestones

Date	Milestone or Go/No-go	Goal description	Status
9/30/2016	Milestone	Develop a high throughput HEXRD-based <i>in-situ</i> characterization process to obtain desired RA volume fraction and stability for 3rd GEN AHSS	Completed
9/30/2018	Milestone	A phase field modeling framework that can predict the effects of heat treatment parameters (i.e., IA temperature and time) on the phase volume fractions as well as C and Mn content in each phase	On track



# Technical Accomplishments and Progress:

- ▶ Alloy systems have been identified for the current studies.
- ▶ PNNL/CSM performed SEM characterization, SEM-EDAX studies of Mn segregation in as-received 7Mn and 10Mn steels.
- ▶ High throughput in-situ HEXRD measurement technique has been developed by PNNL & APS to obtain:
  - Austenite transformation kinetics during IA heating and cooling (H&C) cycles
  - Lattice parameters of austenite and ferrite/martensite during IA H&C cycles.
  - Austenite transformation kinetics during tensile deformation
  - Lattice strains variation during tensile deformation
- ▶ CSM/ASPPRC have performed Dilatometry tests for 7Mn & 10Mn steels using identical thermal cycles as in-situ HEXRD IA cycles.
- ▶ CSM/ASPPRC performed initial ThermoCalc-DICTRA simulations for 5Mn, 7Mn steels for austenite transformation kinetics during IA holding process.
- ▶ CSM/ASPPRC acquired commercial phase field software (MICRESS) through in-kind contribution. More accurate calibration of thermodynamic parameters will be performed with results of in-situ techniques.

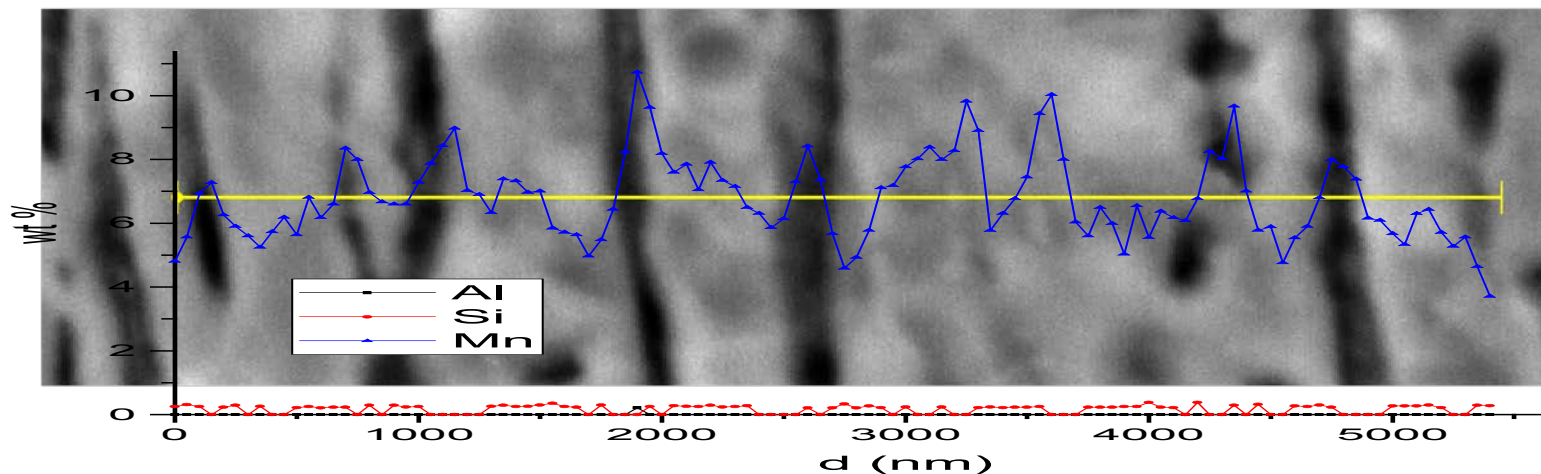
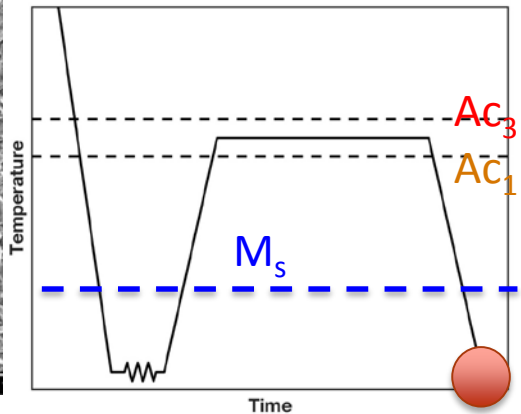
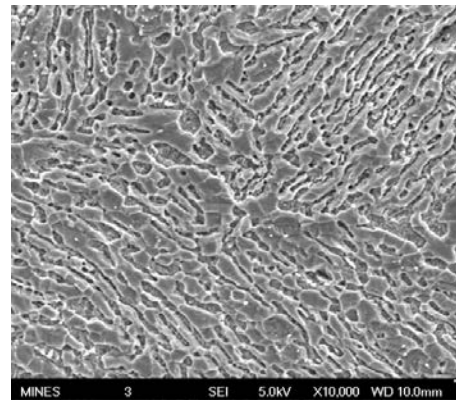
# Technical Accomplishment: Alloy Systems Identified & As-received Materials Characterized (CSM/ASPPRC/PNNL)

- ▶ Steels with 5-10 wt% Mn content are selected in current studies

- ▶ 7 Mn Steel SEM-EDAX Analysis

- Alternating austenite/ferrite lamellar structure observed
- EDX line scan clearly shows high/low Mn phases
- The as-received microstructure has gone through an intercritical annealing (IA) process

0.14C-0.2Si-7Mn

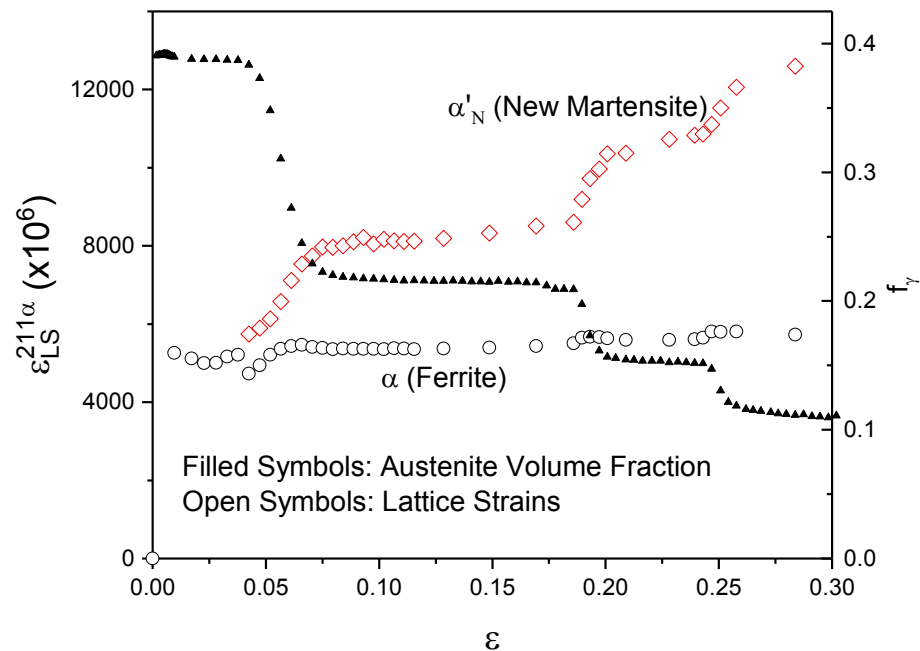
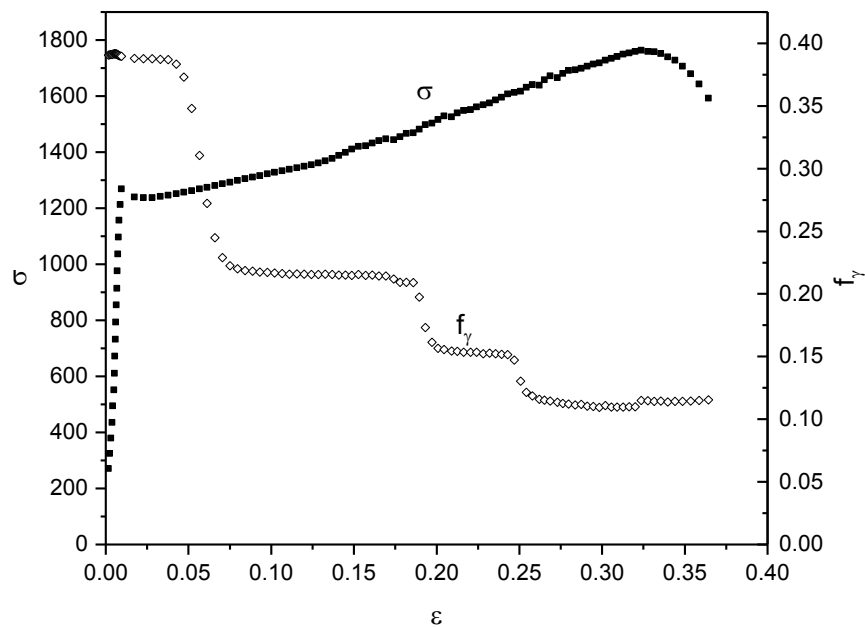




# Technical Accomplishment: In-situ Synchrotron-based High Energy X-ray Diffraction (HEXRD) Tensile Tests (PNNL/APS)

## ► 7Mn steel *in-situ* HEXRD tensile tests were performed at APS

- Step-wise martensitic transformation observed, indicating deformation banding behavior [1]
- Deformation induced transitional phase,  $\epsilon$ -martensite, observed, although very small amount.
- Obtained lattice strains for grains with lattice planes normal to the in-situ tensile loading direction:
  - The 211 lattice strains of ferrite-like phases closely relate to austenite transformation kinetics
- These lattice strains can be used for inverse calculation of individual phase properties [2, 3]

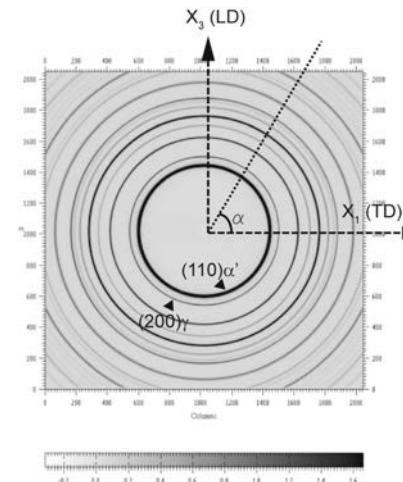


1. Abu-Farha, Hu, Sun, Lu, *Mater Metall Tran A* submitted.
2. Hu, Choi, Sun, Ren, Wang, *Mater Metall Tran A* 2016
3. Hu, Sun, Hector, Ren, *Acta Mater.* 2017

# Technical Accomplishment: In-situ Synchrotron-based High Energy X-ray Diffraction (HEXRD) Tensile Tests (PNNL/APS)

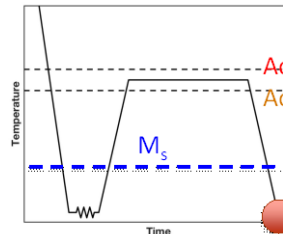
- Identified in-situ heating stage which can be used to perform high throughput thermal cycle experiments under HEXRD beam:

- Linkam TS1500
  - Up to 1500°C
  - Heating rate: 200°C/min
  - Cooling rate: 150°C/min
  - Oxidation prevented with inert gas

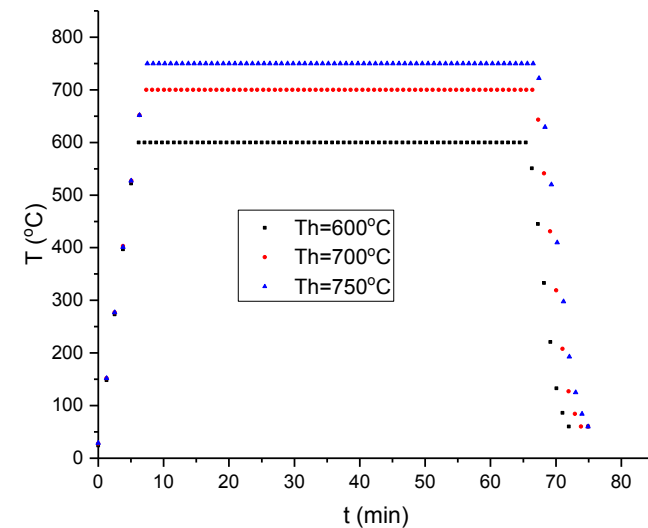


- Performed high throughput in-situ IA experiments on 7Mn steel at different temperatures, different soak times and different cooling rates:

- 600°C, 650°C, 700°C, 750°C
- 60min
- 120°C/min

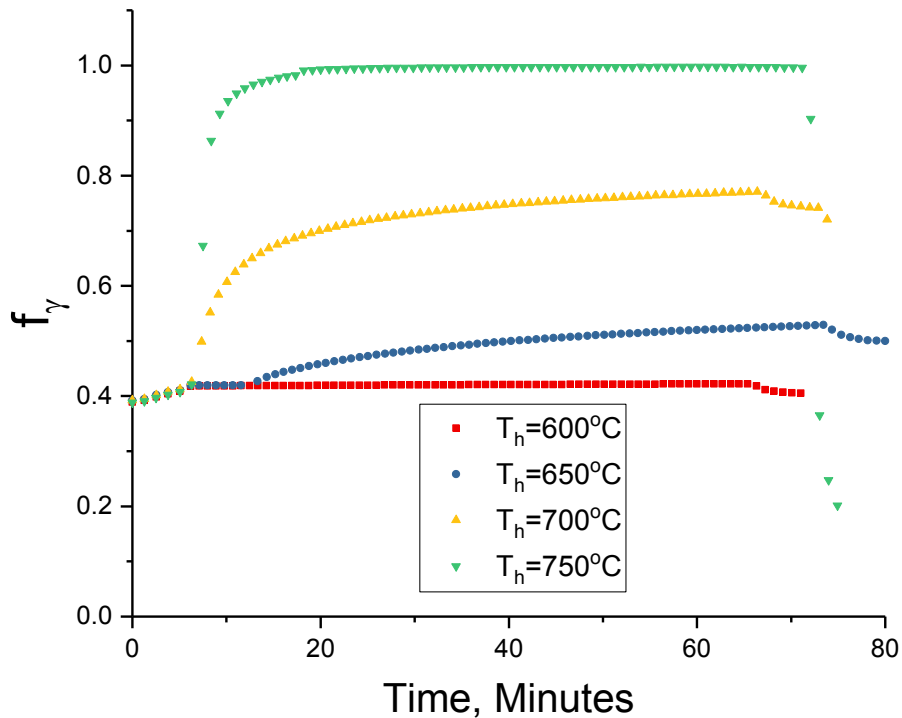


- Obtained in-situ measurements of diffraction patterns and phase volume fraction kinetics during heating and cooling

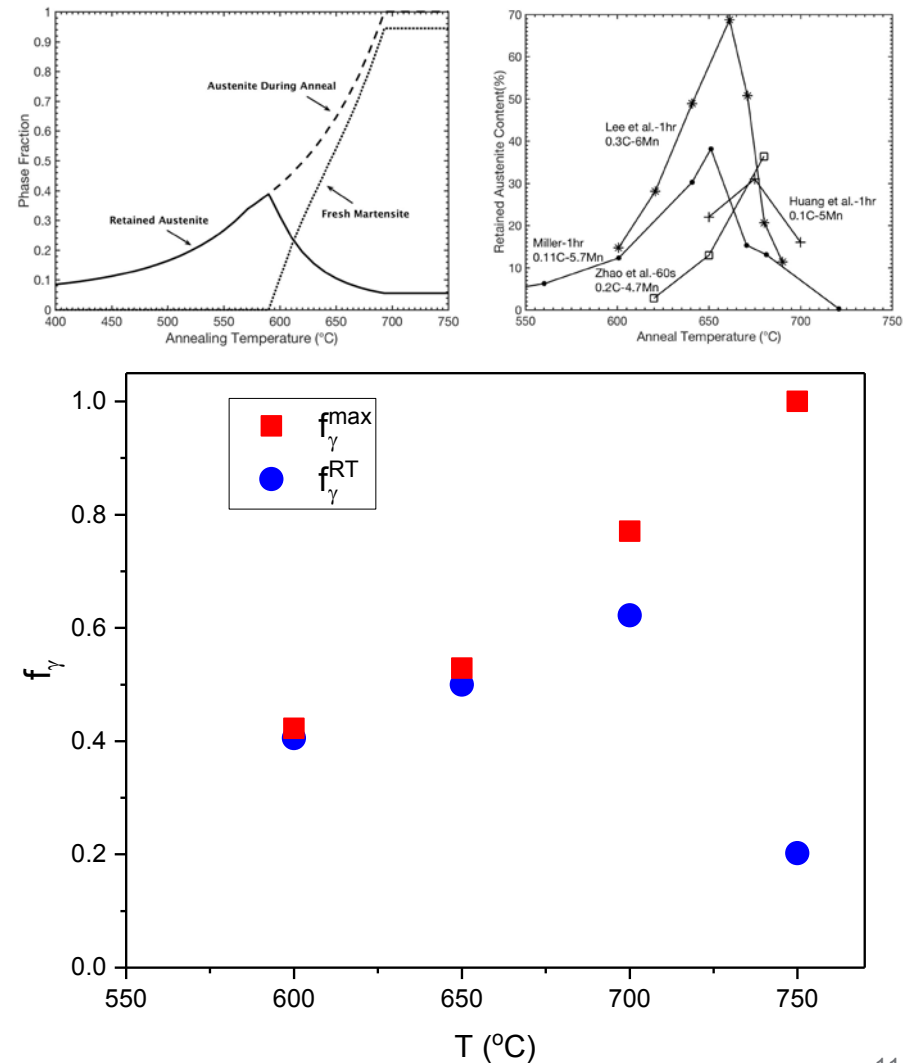


# Technical Accomplishment: In-situ Synchrotron-based High Energy X-ray Diffraction (HEXRD) Tensile Tests (PNNL/APS)

- Measured effects of IA temperature on retained austenite volume fraction (RAVF) evolution



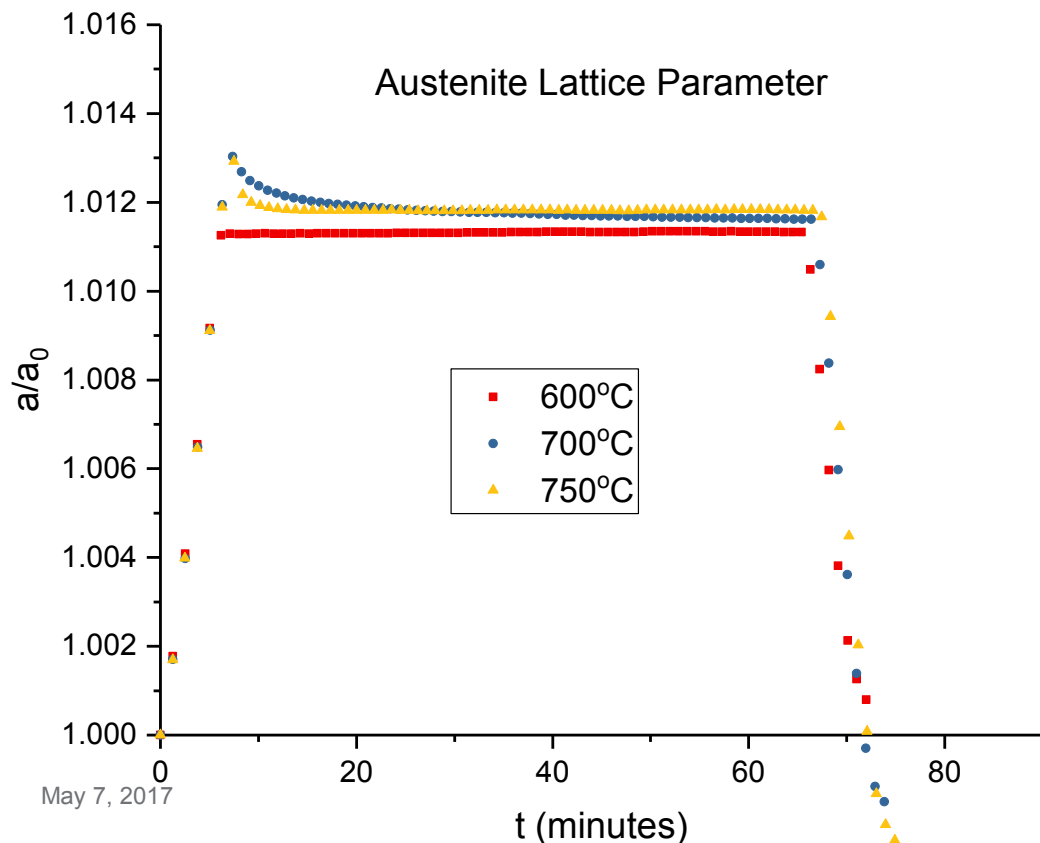
With conventional approach only the RAVF at **RT** can be measured, the *in-situ* HEXRD method can provide RAVF variation with **temperature** and **time**.



# Technical Accomplishment: In-situ Synchrotron-based High Energy X-ray Diffraction (HEXRD) Tensile Tests (PNNL/APS)

## ► Measured lattice parameter evolution

- Heating: Lattice parameters increase rapidly due to thermal expansion
- Holding: Increase slightly with time under 600°C, but decrease for higher temperatures
- Cooling: Decrease rapidly due to thermal contraction

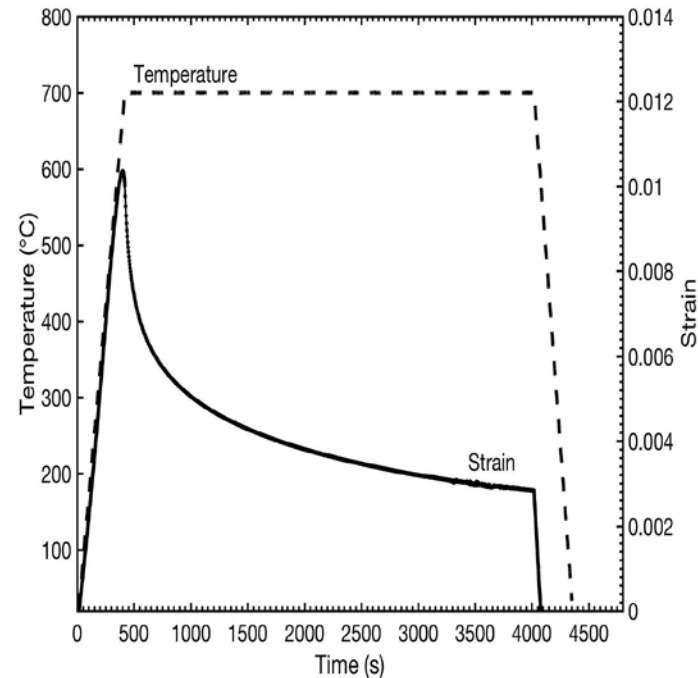
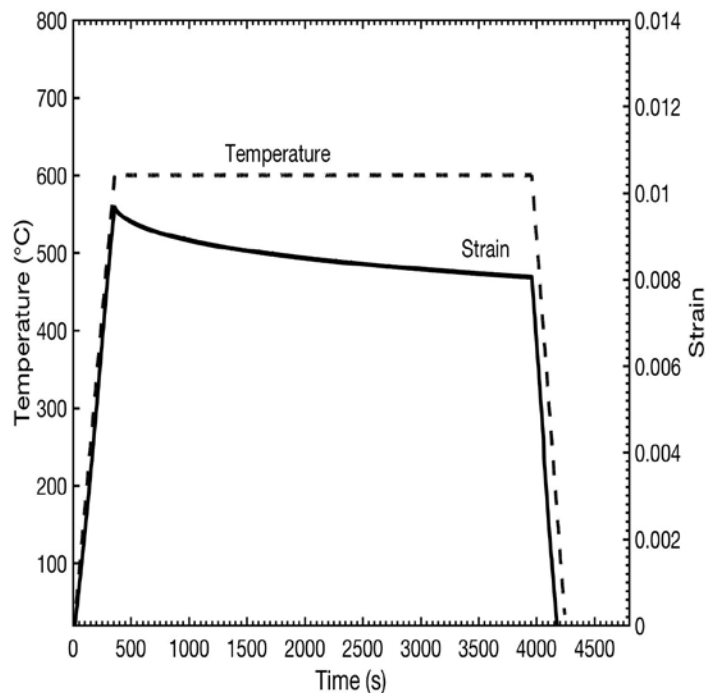


**Linking lattice parameter evolution to diffusion kinetics and phase transformation:**

The lattice parameters will be used to calculate C and Mn content in the alloys during the IA cycles. The variation with temperature will be used as calibration parameters to obtain the thermodynamic parameters in subsequent phase-field modeling.

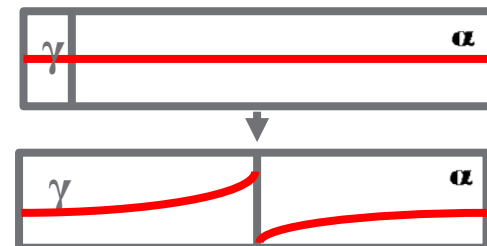
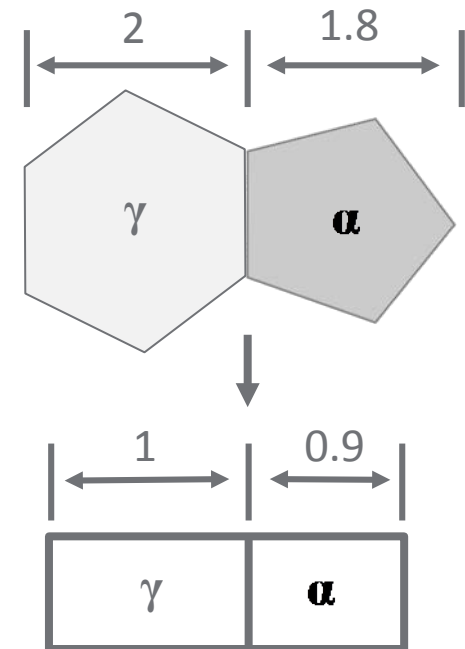
# Dilatometry of 7Mn Steel (CSM/ASPPRC)

- Dilatometry studies are performed using the identical thermal cycles with those of in-situ HEXRD thermal tests



Dilatometry measures mainly volume change during phase transformation, therefore shows much larger strain than the variation of lattice parameters from in-situ HEXRD experiments.

- ▶ ThermoCalc-DICTRA models have been conducted to simulate the IA holding process
  - One-dimensional model
  - Rely on mobility and thermodynamic databases
  - Cell size: Half width of grains
  - Mobile interfaces
  - Example problem
    - Composition: 0.2C-5Mn
    - Starting microstructure assumed to be the one that is cooled down to 25 °C after full austenization
      - ◆ Fraction Martensite: 0.953 (Koistinen-Marburger)
      - ◆ Fraction Austenite: 0.047
    - ThermoCalc-DICTRA model simulates the transformation when the above starting microstructure of 5Mn steel holding at 650°C.
    - Cell size influences are studied initially.



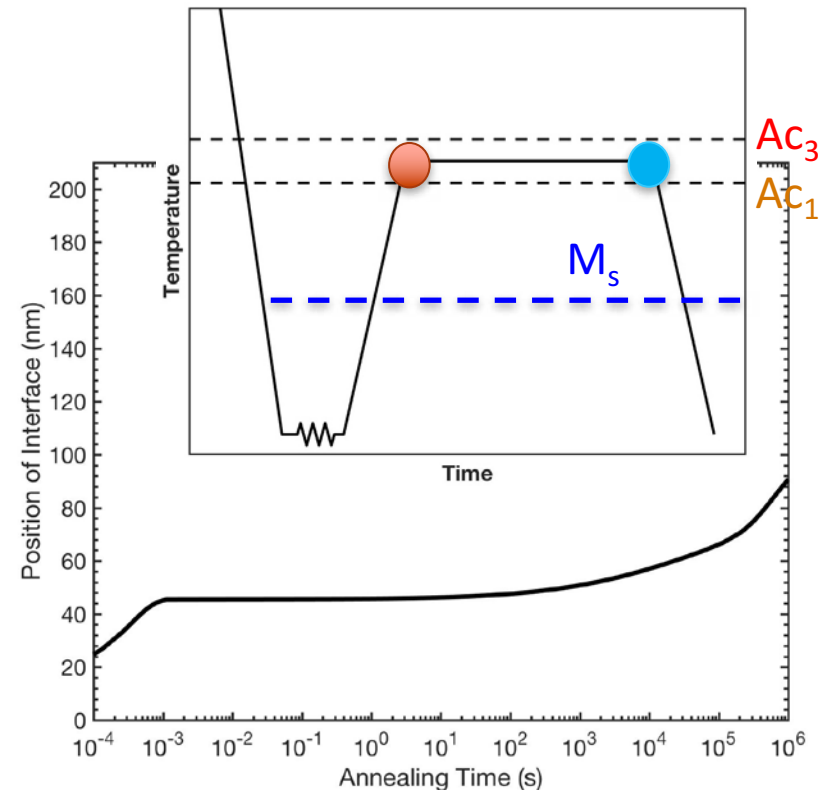
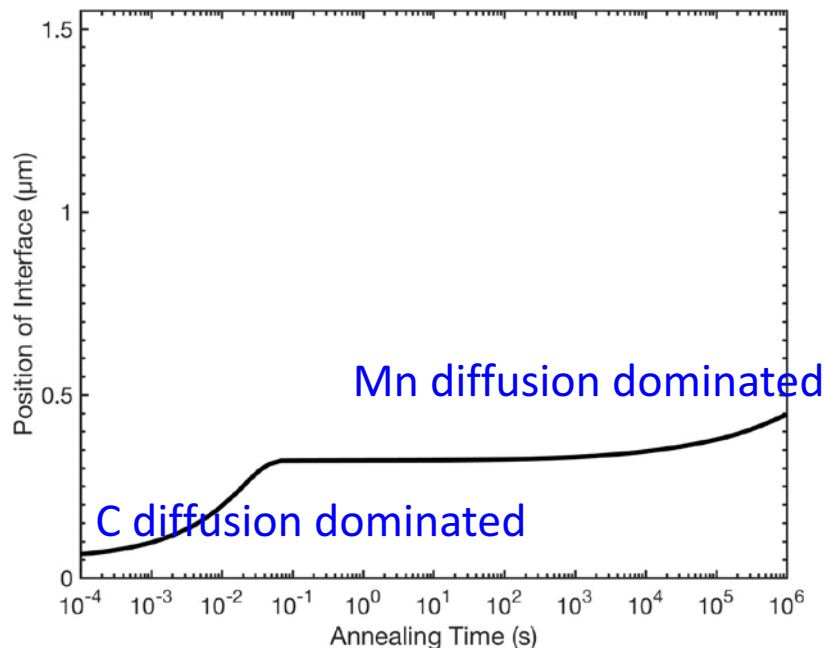


# Technical Accomplishment: ThermoCalc-DICTRA Simulations: Effects of Cell Size (CSM/ASPPRC)

- ▶ Cell size influences on transformation are examined
  - The starting microstructure has very little austenite. Heating and cooling stages are not considered.
  - Smaller cell size shows much shorter period of carbon diffusion dominated transformation.
  - Future studies will include simulations of the full *in situ* HEXRD IA thermal cycle with as-received material as starting microstructure (Any proposed future work is subject to change based on funding levels).

Initial Cell Sizes:  $\gamma$ -10 nm  $\alpha$ -200 nm

Initial Cell Sizes:  $\gamma$ -0.05  $\mu\text{m}$   $\alpha$ -1.5  $\mu\text{m}$



# Responses to Previous Year Reviewers' Comments

## ► Approach to performing the work

- The reviewer said that primarily literature study is presented, validation data would be helpful.
- Response: The project just started at last AMR time. This year, much more progress on experimental and simulation are presented.
- The reviewer said picking judicious methods in Thrust 4 is not defined. The reviewer commented that this plan for improvements seems too vague for a robust approach, and that the project team is using too many acronyms that are not generally well known. The reviewer noted that HEXRD, RA, ASPPRC, APS, IA, TRIP, TOF-SIM, and CCE are only known to material scientists, and asked that the project team please define these the first time they are used in slides or reports.
- Response: In this presentation, the use of acronyms is reduced or it is defined the first time they are used. More experimental results necessary for the full understanding of the mechanistic behavior of medium Mn steel during tensile deformation. This will influence the method in modeling such behavior.

# Responses to Previous Year Reviewers' Comments

## ► Technical accomplishments and progress toward overall project and DOE goals

- The reviewer said that this project has just started and there are few accomplishments at this time. How the experimental results will be used in the ICME models is unclear to this reviewer and has not been defined in the presentation.
- Response: This is not an ICME project. Rather, it focuses on the specific length scale of diffusion and phase transformation in order to accelerate the development process of 3<sup>rd</sup> GEN AHSS from the processing perspective.

## ► Collaboration and coordination with other institutions.

- There is not a clear division of labor or clear roles and responsibilities. There is an assembled team, but the project would be improved if the assignments to each member were more clear and distinct. The reviewer commented that where the experiments will be done and who will be doing the math modeling should be clearer
- Response: In this presentation, the roles and responsibilities of different teams are made more clear.

# Responses to Previous Year Reviewers' Comments

## ► Proposed future research

- The reviewer said that the presentation does not have a specific slide to show the future work, but it can be seen from the tasks listed in Slide 5. The reviewer said that it is good to have the future work that will be done before the next Annual Merit Review meeting listed in a separate slide.
- The reviewer said that there is no definition of the next steps in the developments. The reviewer understands there will be testing, but the reviewer saw no details on how the specimens will be developed, or what will be done with the test results.
- Response to both reviewers: A separate slide of future work is added to the current presentation.

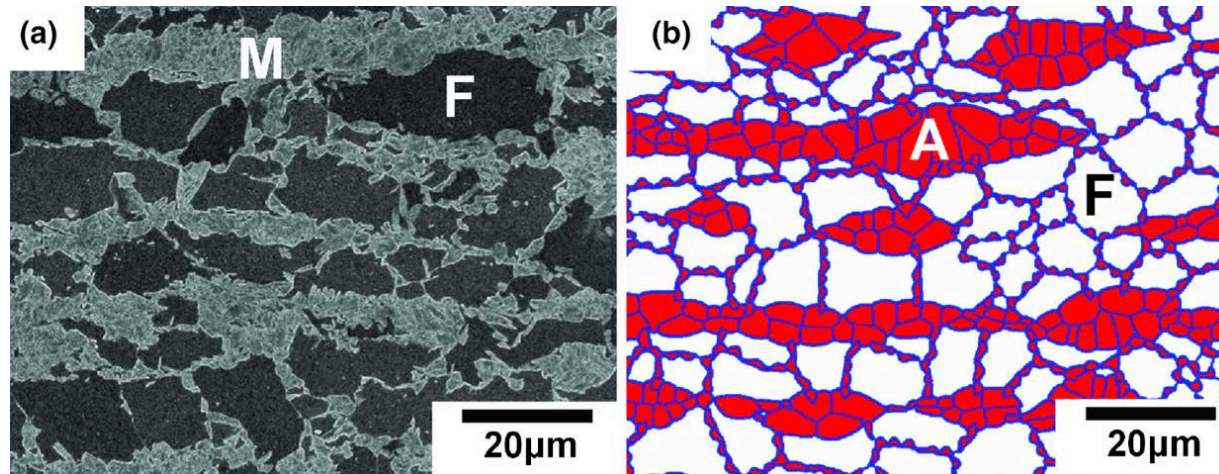
- ▶ **Task 1: Conventional Experimental Characterization (ASPPRC)**
  - More systematic parametric studies are necessary for the traditional thermal treatment (Austenization + IA thermal cycles) to examine their influences on C and Mn partitioning between phases.
  - Tensile testing of the resultant materials from above with digital image correlation (DIC) and microstructural examination needs to be performed to understand the banding behavior and yield point elongation
- ▶ **Task 2: Advanced HEXRD in-situ Characterization (PNNL/APS)**
  - A full analysis of transformation kinetics and lattice strain changes with identical thermal cycles from task 1 is needed to obtain more complete transformation kinetics data for validation of thermo-dynamic parameters in Task 3.
  - In-situ HEXRD tensile testing of resultant materials (with DIC if possible) are necessary to fully understand the deformation mechanism.
- ▶ **Task 3: Microstructural Evolution and Deformation Modeling**
  - Parameters in microstructural evolution modeling techniques (ThermoCalc-DICTRA /MICRESS phase field) will be further validated and calibrated with identical experiments from Tasks 1 and 2 (CSM/ASPPRC):
  - Based on results of ex-situ and in-situ mechanical test and analysis results, mechanistic models need to be established to accurately predict the tensile behaviors of the median Mn steels. (PNNL)

- ▶ **Task 1: Traditional Experimental Characterization (ASPPRC)**
  - Austenization (AUS) followed by intercritical annealing (IA) thermal treatments with different set of parameters will be performed
  - SEM-EBSD, TEM-EDAX microstructure examination of the resultant materials from above to examine RT austenite volume fraction, C and Mn distribution between phases.
  - Tensile tests of the resultant materials with digital image correlation (DIC) and microstructural examination (SEM-EBSD, TEM) will be performed to gain full understanding of the banding behavior and yield point elongation
- ▶ **Task 2: Advanced HEXRD in-situ Characterization (PNNL/APS)**
  - Perform a full analysis of transformation kinetics and lattice strain changes with identical thermal AUS+IA cycles from task 1. The results of both task 1 and 2 will be used to validate and calibrate thermodynamic parameters for modeling in task 3.
  - In-situ HEXRD tensile testing of resultant materials (with DIC if possible) to fully understand the deformation mechanism.
- ▶ **Task 3: Microstructural Evolution and Deformation Modeling**
  - Establish and perform model simulations of microstructure evolution using ThermoCalc-DICTRA / MICRESS utilizing identical AUS+IA thermal cycles of tasks 1 and 2. With the results of tasks 1 and 2, the thermodynamic parameters will be validated and calibrated from the models (CSM/ASPPRC).
  - Establish mechanistic models to accurately predict the tensile behaviors of the medium Mn steels, based on results of ex-situ and in-situ mechanical test and analysis results (PNNL).



# Future Works: MICRESS Phase Field Modeling

- ▶ Inputs from experimentation
  - Initial cell size
  - Mobility
  - Solute availability (cementite precipitation/dissolution)
  - $M_s$
- ▶ Generate phase field model using MICRESS®



Dual-Phase steel: a) observed room-temperature microstructure, b) phase field model during intercritical anneal [1].

1. Militzer, *Current Opinion in Solid State and Materials Science*, 2011.

# Collaborations and Progress Summary

- ▶ Task 1. Traditional experimental characterization (CRM/ASPPRC/PNNL):
  - SEM-EDAX characterization of as-received 7Mn and 10Mn steels.
  - Dilatometry experiments performed with identical thermal cycles of in situ HEXRD thermal experiments.
- ▶ Task 2. Advanced HEXRD in-situ characterization (PNNL/APS):
  - IA heating methodology developed and tested
    - In-situ measured RA volume fraction and lattice parameters
  - In-situ tensile testing have been performed with retained austenite volume fraction evolution with strain analyzed.
- ▶ Task 3. Thermodynamic modeling of phase transformation (CSM/ASPPRC) and Linking microstructures to properties (CSM/PNNL):
  - One-dimensional DICTRA™ simulation of phase transformation of 5Mn steel has been performed.